VOLATILE ORGANIC COMPOUNDS MONITORING NETWORK

QUALITY ASSURANCE PLAN

MARCH 1990

Ontario
Environment
Environnement
Jim Bradley, Minister/ministre

ISBN 0-7729-6595-1

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QUALITY ASSURANCE PLAN

Report prepared by:

Special Studies and Research Management Unit
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Air Resources Branch

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1.0 Scope and Purpose of the Quality Assurance Plan

Airborne chemicals exist in particulate and vapour and organic and inorganic forms. Organic compounds are of particular concern due to their complexity and variety of potential toxic effects, including carcinogenicity. Over the past few years the Organics Monitoring Sub-Committee of the Ambient Air Quality Monitoring Committee (formerly the Organic Vapour Sampling Committee) of the Ontario Ministry of the Environment has developed methods for measuring a number of volatile organic compounds (VOCs) in ambient air. The finalized measurement method is now being incorporated into a provincial network the results from which will provide a sound basis for hazard assessment and control action.

The purpose of the network is to determine quantitatively ambient air levels of volatile organic compounds at a number of urban sites in Ontario. Several background, or non-urban sites are incorporated for comparison purposes. The specific objectives of the program are:

1) To monitor ambient levels of targeted volatile organic compounds at selected urban and rural sites in Ontario to assess health impacts on the population.
2) To determine monthly, seasonal, and annual trends of volatile organic compound concentrations in the province of Ontario to assess the effectiveness of abatement programs.

Benefits of a province-wide VOC monitoring network include 1) comparison of typical urban and rural sites; 2) identification of problem areas based on anomalously high VOC concentrations; 3) generation of a historical database for ambient air which may later be used in conjunction with personal monitoring and indoor air monitoring to characterize the total inhalation exposure of the population of Ontario; and 4) comparison with other agencies for equivalency.

Inherent in ambient air monitoring network operations is the commitment to ensure that all data are scientifically valid, defensible, thoroughly documented and reported with quality indicators. To achieve this goal a quality assurance program, i.e. "a system for integrating the quality planning, quality assessment and quality improvement efforts of various groups in the organization to enable operations to meet user requirements at an economical level" (U.S. EPA, 1976), is considered to be a crucial element of the volatile organic compound monitoring program and has thus been integrated into the network operations to enhance data credibility. The Ministry's central laboratory has implemented its own extensive quality assurance program which is managed as an internal laboratory function (OME, 1989a). Only a brief synopsis of the laboratory quality assurance requirements which interface with the volatile organic compound measurement program will be given here.

The term quality assurance (QA) is a general one and encompasses Quality Control (field, laboratory, and data activities) and Quality Assessment (performance

check and audit activities). These activities are shown as a simplified flow chart in Figure 1.1 (Oslund, 1986).

Figure 1.1. Quality Assurance for Ambient Air Volatile Organic Compound Monitoring Network (adapted from Oslund, 1986).

Proper implementation of the quality assurance program relies on two documents whose rationale and quality assurance guidelines and activities augment and complement existing standard operating procedures and which help to minimize factors that could negatively affect data quality. These documents are:

1) QA Program Plan - "an orderly assembly of management policies, objectives, principles, and general procedures by which an agency or laboratory outlines how it intends to produce quality data" (U.S. EPA, 1976).

2) QA Project Plan - "an orderly assembly of detailed and specific procedures by which an agency or laboratory delineates how it produces quality data for a specific project or measurement method" (U.S. EPA, 1976).

This document describes only the quality assurance program plan (hereafter called the QA Plan). The quality assurance project plan (hereafter called the QA Manual) should be referred to for the detailed quality control and quality assurance procedures which have been designed to meet the objectives stated in this document. Information regarding network operation is provided in the Standard Operating Procedures and Technical Manual (OME, 1989b).

2.0 Components of the Quality Assurance Plan

The function of the QA Plan is threefold. Firstly, it identifies the operational components within the VOC monitoring program which require quality assurance (generally separated into Field Operations, Laboratory Operations, and Data Management Operations). Secondly, the QA Plan states the quality assurance objectives for each of the network components and explains how each component contributes to achieving the overall QA Plan objective. Thirdly, the QA Plan describes the structural organization and resources, the data gathering and reporting activities and responsibilities, and the control systems and contingency procedures required to ensure that the overall QA Plan objective is met.

The overall QA Plan objective is:

> to ensure that the data quality characteristics of accuracy, precision, completeness, representativeness, and comparability are attained to given levels of acceptability through the design, description and implementation of quality control and quality assessment guidelines and procedures.

The data quality characteristics of accuracy, precision, completeness, representativeness, and comparability and their acceptance criteria are defined below.

1) Accuracy: "the degree of agreement of a measurement, X, with an accepted reference or true value, T, usually expressed as the difference between the two values, X-T, or the difference as a percentage of the reference or true value, 100(X-T)/T, and sometimes expressed as a ratio, X/T" (U.S. EPA, 1976). Lack of accuracy indicates absolute bias; i.e. a systematic (consistent) error between test results and the true value.

Accuracy can not be measured in the field as there is no absolute standard (i.e. a known volume and composition of VOC) with which to challenge the monitoring equipment. Accuracy is indirectly inferred by assuming that VOCs are homogeneously distributed within the volume of air presented to the sampler and that the volume of air can be accurately measured by reference to a primary standard. For the sampler to be considered "accurate" (given the above constraint) the volume of air determined by the VOC sampler must agree with that determined by the primary standard to within ± 10%.

Analytical accuracy is readily determined using standard reference materials (SRMs) obtainable from the U.S. National Bureau of Standards or commercial operations. The volatile organic compound concentrations determined by the Ministry's laboratory must agree to within ± 10% of the values stated on the SRM's certificate of analysis to be considered accurate.

2) Precision: "a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is most desirably expressed in terms of the standard deviation but can be expressed in terms of the variance, range, or other statistic. Various measures of precision exist depending upon the "prescribed similar conditions."" (U.S. EPA, 1976).

It should not be assumed that VOC measurements taken by a particular instrument at a specific site are reproducible over time. It is therefore necessary to conduct "precision" testing to determine the reproducibility of the measurement process. As it is not possible to collect simultaneous VOC samples using the same instrument, a second, collocated, sampler of the same type is used to assess the reproducibility, or precision, of the measurement. The precision of volatile organic compound concentrations from collocated samplers is, in large part, dependent upon the analytical precision associated with each compound.

Acceptable levels for analytical precision are determined by the Ministry laboratory's Quality Control/Quality Assurance Office. For VOC analysis, an overall analytical precision is impractical as the precision varies with the volatility of the compound being analyzed. For this reason, the analytical precision for each compound is reported with the results.

3) Completeness: "the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal operations, usually expressed as a percentage" (U.S. EPA, 1976).

Although a perfect rate of data capture is ideal for any monitoring program, such unavoidable sampling and analytical problems as instrumentation malfunction and down-time lead to data capture rates less than 100%. VOC data are to be considered complete and representative of the sampling effort for a particular site if ≥ 75% of the possible annual observations yield valid (see § 4.3) VOC concentration estimates. This annual completion rate must be qualified as it is quite possible to attain the 75% target without truly representing a particular season or seasons.

Of the possible seasonal observations, ≥ 67% must yield valid volatile organic compound concentration estimates. This target is independent of how many seasons a sampling year may be divided into: e.g. winter and summer only or winter, spring, summer, and fall.

4) Representativeness: the degree to which data "represent[s] a lot or population as accurately and precisely as possible" (U.S. EPA, 1976).

Representativeness is a term that can be used to desc e various sources of error in VOC measurements. It is a function of two potential sources of error: siting problems; and sampling problems. Sampling problems are best assessed in terms of precision, accuracy, and completeness and thus "representativeness" will focus on siting only.

The sampling network is designed to assess ambient levels of volatile organic compounds at urban and non-urban sites. Sites must be located in areas representative of the conditions being measured (i.e. urban sites must be located in typical urban settings; non-urban sites must be located in typical non-urban settings) and must be free of local interferences. In an urban situation, VOC concentrations should be typical of those within a 1 km radius of the sampling site. In a non-urban situation where background conditions are being sampled, VOC concentrations should be typical of those within a 10 km radius of the sampling site.

Representativeness is addressed initially in the site selection criteria and evaluation (see QA Manual). Continuous observation of land use and development is to be documented throughout the life of the sampling program and periodic external evaluation of a site's representativeness is to be done as part of a system audit.

5) Comparability: "A measure of the confidence with which one data set can be compared to another." (U.S. EPA, 1976).

Comparability is also a general term as it implicitly involves the assessment of accuracy and precision. The most familiar assessment of comparability is the degree to which data sets obtained during inter-agency intercomparison studies are comparable. These data sets will be considered comparable if statistical tests indicate there is no significant difference at a given confidence level (usually 95%). In order to test comparability, data must be in consistent units (e.g. $\mu g/m^3$).

Comparability also includes the analytical determination. The analytical method of determination must be shown to be the most feasible and should be comparable to equivalent methods of determination.

3.0 Organization and Responsibilities

The Volatile Organic Compound Monitoring Network is a co-operative venture involving the Air Resources Branch (ARB), the Laboratory Services Branch (LSB), and the regional operations (Southwestern - SW; Southeastern - SE; Northwestern - NW; Northeastern - NE; West Central - WC; and Central - C) of the Ministry of the Environment. The network is guided by the Organics Monitoring Sub-Committee of the Ambient Air Quality Monitoring Committee and is administered by the Atmospheric Research and Special Programs section of Air Resources Branch. The sub-committee is comprised of representation from the regions (2), the laboratory, and Air Resources Branch and is chaired by the Air Quality Chief from one of the regions.

Resources and support for the Volatile Organic Compound Monitoring Network are as follows:

- Overall program coordination, network QA management, field operations management, training, instrumentation development, data management and reporting, and contact with other agencies (etc.) are the responsibilities of the Air Resources Branch.

- Laboratory operations including cartridge preparation and proofing,

sample reception, sample analysis, and analytical data reporting are the responsibility of the Laboratory Services Branch.

- Regional operations provide any manpower and technical assistance necessary to carry out monitoring activities including sample collection, shipping, and routine maintenance and calibration of the sampler.

Decisions relating to the scientific direction, policy-making and progression of the Volatile Organic Compound Monitoring Network are to be determined by consensus of the Organics Monitoring Sub-Committee at regularly scheduled meetings. It is the responsibility of the "Network Coordinator" (ARB) to ensure that the decisions of the Sub-Committee are implemented in a timely fashion. The minutes of Sub-Committee meetings are to be distributed to its members for information purposes and as a follow-up to items requiring action. Sub-Committee members are to disseminate this information to their subordinates, where appropriate. In the interval between Sub-Committee meetings, discussions of policy matters will be communicated to and from the Network Coordinator. The minutes are also to be distributed to the Ambient Air Quality Monitoring Committee.

An organizational chart for the Volatile Organic Compound Monitoring Network is shown in Figure 3.1. This chart establishes the locations of QA

responsibilities, the lines of communication, and the internal flow of QA information. A brief summary of QA responsibilities is also provided.

The success of the QA program and the sampling program in general depends on the following organizational requisites being met:

i) Sufficient manpower must be made available to carry out the work specified, within time and funding constraints.

ii) All personnel must know and understand the QA responsibilities of their positions and must possess the necessary education, training, experience, and commitment to fulfil their QA-related duties.

iii) Personnel qualifications, training needs, and QA requirements as they relate to the Volatile Organic Compound Monitoring Network are to be identified and described in detail in the QA Manual.

iv) Quality assurance reports (reports of data quality to data users and quality assurance reports to management) are to be issued regularly, preferably on an annual basis. These reports are to be reviewed by the Organics Monitoring Sub-Committee. Any follow-up actions identified in these reports are to be duly acted upon by the Network Coordinator.

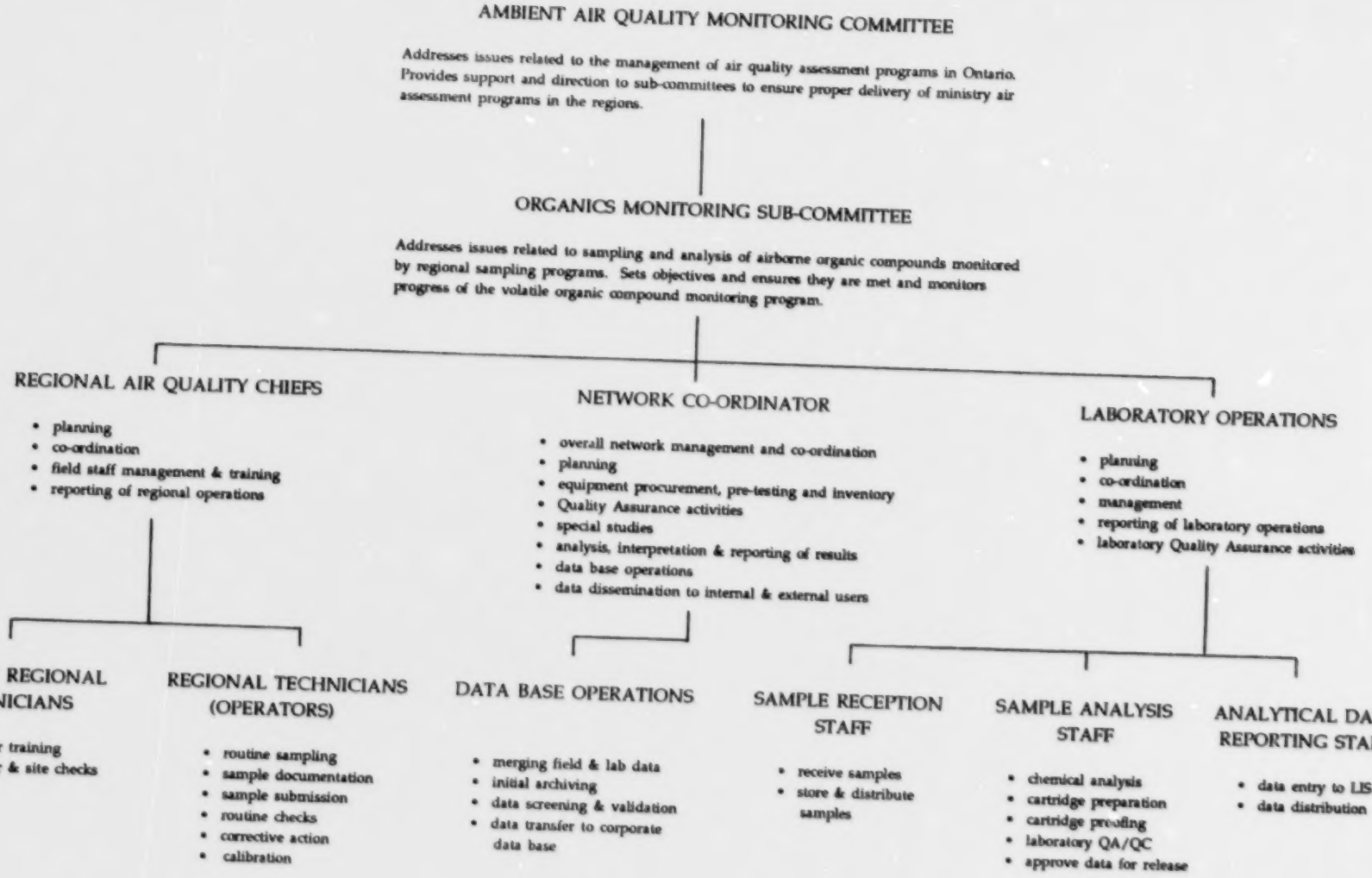
AMBIENT AIR QUALITY MONITORING COMMITTEE
Addresses issues related to the management of air quality assessment programs in Ontario. Provides support and direction to sub-committees to ensure proper delivery of ministry air assessment programs in the regions.
ORGANICS MONITORING SUB-COMMITTEE
Addresses issues related to sampling and analysis of airborne organic compounds monitored by regional sampling programs. Sets objectives and ensures they are met and monitors progress of the volatile organic compound monitoring program.
REGIONAL AIR QUALITY CHIEFS
• planning
• co-ordination
• field staff management & training
• reporting of regional operations
NETWORK CO-ORDINATOR
• overall network management and co-ordination
• planning
• equipment procurement, pre-testing and inventory
• Quality Assurance activities
• special studies
• analysis, interpretation & reporting of results
• data base operations
• data dissemination to internal & external users
LABORATORY OPERATIONS
• planning
• co-ordination
• management
• reporting of laboratory operations
• laboratory Quality Assurance activities
SENIOR REGIONAL TECHNICIANS
• operator training
• operator & site checks
REGIONAL TECHNICIANS (OPERATORS)
• routine sampling
• sample documentation
• sample submission
• routine checks
• corrective action
• calibration
DATA BASE OPERATIONS
• merging field & lab data
• initial archiving
• data screening & validation
• data transfer to corporate data base
SAMPLE RECEPTION STAFF
• receive samples
• store & distribute samples
SAMPLE ANALYSIS STAFF
• chemical analysis
• cartridge preparation
• cartridge proofing
• laboratory QA/QC
• approve data for release
ANALYTICAL DATA REPORTING STAFF
• data entry to LIS
• data distribution

Figure 3.1. Organizational Chart

4.0 Network Operations

The overall emphasis on quality assurance is focused on the network operations of the Volatile Organic Compound Monitoring Network. "Network Operations" includes Field Operations; Laboratory Operations; and Data Management Operations, each of which is described below in detail.

The integrity of all samples will be assured provided:

i) well-conceived, scientifically-sound standard operating procedures and QA protocol are rigorously complied with by all personnel at all times;

ii) all personnel are thoroughly trained to carry out their respective duties and documentation of these duties is readily available.

iii) dependable, state-of-the-art instrumentation is used in the network and preventative and remedial maintenance procedures are established to minimize down-time.

4.1 Field Operations

Accuracy, precision, completeness, representativeness, and comparability are most sensitive to field operations. Field operations are the least controllable area of network operations because they are carried out by a large number of people across the province.

Field operations address the following:

i) Network representativeness

ii) Field instrumentation

iii) Sample collection and handling

4.1.1 Network Representativeness

To ensure network representativeness, monitoring stations are to be selected such that the geographical distribution of sites meets the monitoring network's objectives. Sites selected for monitoring must receive volatile organic compound concentrations representative of the surrounding area. For all sites, general siting criteria (see QA Manual) are to be followed (e.g. avoidance of topographical anomalies, obstructions etc.). Any deviations from acceptable criteria are to be documented and data collected from these sites are to be closely scrutinized.

Ensuring that the site distribution meets its network objectives is the responsibility of the Organics Monitoring Sub-Committee which must review the available data and make an assessment of the site distribution for its suitability.

An essential element to assess representativeness is the documentation used to describe and evaluate all sites. A comprehensive listing of site selection criteria and a site description is to exist for every site. The Network Coordinator is to incorporate this information into a standardized site description form (see QA Manual). The form is a modification of the National Air Pollution Surveillance site documentation (Environment Canada, 1988). The site description must include the following:

i) location of the site including station name, station number, address, latitude, longitude, UTM co-ordinates (Northing and Easting) and elevation above ground and above sea level. An area map (street or topographic) must be kept on file for each site;

ii) the start-up date of the station;

iii) the name, address, and telephone number of the regional technician responsible for the site and of any alternates;

iv) scale of representativeness (i.e. neighbourhood, regional, etc.) and land use (i.e. industrial, commercial, residential, rural, etc.);

v) a description of how to travel to the site and any other access information;

vi) a description of the electrical service at the site;

vii) a listing of all VOC equipment installed at the site used for sample collection, including the name of manufacturers, model and serial numbers;

viii) a description of the surrounding topography and a list of other site influences;

ix) a description of human and vehicular activities near the site;

x) a site diagram indicating the position of the sampling manifold, the location and type of surrounding buildings, roads, ground cover, obstructions, potential contamination sources in the immediate vicinity of the manifold inlet, electrical installations and compass points. The diagram is to be drawn to scale;

xi) location of stationary pollutant sources including distances and directions from the site. Descriptions of any other potential sources are to be included in this documentation;

xii) 4 site photographs are to be taken in the direction of the 4 cardinal points. The photos should clearly show the manifold inlet and its surrounding area;

xiii) a summary of the site including advantages, disadvantages, and any deviations from siting criteria.

Monitoring sites may change over time. To determine whether existing sites meet siting criteria, a re-evaluation of each site is to be undertaken during an external QA systems audit. This re-evaluation is to consist of updating site documentation, including re-photographing all sites. After the re-evaluation a report reassessing the suitability of each site is to be submitted to the Organics Monitoring Sub-Committee for review.

Regional personnel are to report immediately to the Network Coordinator any site changes that occur between evaluations. A Site Change Notification form (see QA Manual) is to be completed by the regional technician to document changes made to the site or its surroundings. The effect of any changes on the

quality of the data collected is to be evaluated as soon as possible by the Network Coordinator.

4.1.2 Field Instrumentation

The instrumentation used for sample collection has potential for negatively affecting data quality since an instrument's capabilities and performance can affect accuracy, precision, and completeness. The instrumentation used in a supporting function (e.g. calibration equipment, sample shipment and storage) can also affect data quality.

The instrumentation used in the field operations must be capable of meeting the following objectives:

i) the air sampler must be capable of collecting sufficient volume for the chemical analysis of desired compounds of interest with minimal breakthrough and with the least possible level of contamination or interference;

ii) automated samplers requiring minimal attendance in the field are preferred;

iii) the sampling flow rate must be calibrated to accepted standard methods with the highest possible accuracy;

iv) the instruments to be used for sample collection must be free of any environmental conditions which could negatively affect data quality (e.g. temperature, humidity or moisture build-up, dust levels, etc.);

v) to preserve the chemical integrity of collected VOC samples, sorbent tubes are to be isolated inside shipping containers which must be constructed so as to minimize the possibility of breakage during shipping and handling.

The sampler preferred for use is a collection of off-the-shelf components assembled into a package which satisfies the objectives stated above (OME, 1989b). The components of the sampling train are to be operated according to their design specifications outlined in their instrument manuals (which are to be filed in a binder and kept with other program documentation). To ensure that instruments operate according to their design specifications, the following four quality assurance steps are to be implemented: i) procurement and pre-testing; ii) calibration; iii) preventive maintenance and routine checking; and iv) corrective action.

Procurement procedures, obviously, can only be applied to new samplers acquired for the network. When additional samplers are required every effort should be made to obtain the same type and model of sampler being used in the network. This will ensure continuity and ease of operation of the network. If an equivalent sampler (rather than one identical to what is in current use) must be purchased, the following minimum procurement procedures are to be followed:

i) detailed specifications of required instrument performance characteristics and manufacturing details (e.g. operating tolerance) are to be provided in the purchase order contract;

ii) detailed warranty specifications should be stated in the purchase order. Detailed operating, maintenance and service guidelines, as well as schematic and mechanical drawings should be made available upon purchase;

iii) upon receipt of an instrument, careful inspection is to be done to ensure that all specifications have been met. All equipment is to be tested by Air Resources Branch for acceptance prior to deployment in the field. Procedures for acceptance testing are to be described in the Standard Operating Procedures and Technical Manual.

The nature of the sampling equipment is such that it is unnecessary to maintain an extensive inventory of spare parts. Such spares as will be required from time to time (e.g. glass sampling ports, Tygon tubing, etc.) are to be kept at Air Resources Branch and distributed as requested by the regions.

Flow rate calibrations represent a QA activity for measuring and controlling the accuracy of the network monitoring equipment. A calibration plan with recommended intervals for calibrating measurement instrumentation is to be developed and documented in the QA Manual. All samplers must be within an acceptable calibration range (as specified in the Standard Operating Procedures and Technical Manual) prior to installation and operation at the sampling site. After calibrations have been carried out, the results are to be recorded on calibration log sheets, copies of which are to be submitted to the Network Coordinator as soon as possible. Upon review by the Network Coordinator, any significant changes in accuracy must be accounted for in the data entering the database. As a check on the calibration procedures, independent calibration audits are to be performed periodically by Air Resources Branch and as part of any external performance audit. External auditors are to verify that the audit results have been properly incorporated into the network database.

Increased sampler reliability will result if preventive maintenance and routine checking procedures are carried out on a regular basis. Sampler down-time

is minimized thus increasing data completeness and accuracy. Simple routine checks (e.g. examining tubing for signs of aging or cracking) should be carried out periodically. As problems are identified they can be corrected before instrument failure causes a lost sample. Preventive maintenance and routine checking procedures are to be documented in the QA Manual. Any changes in an instrument are to be recorded in the instrument's log book.

A corrective action plan should be documented in the QA Manual. The success of such a plan depends upon the early detection of problems (through routine checking) and the response time taken by technical staff to rectify the problem. Once a problem has been detected it should be remedied on site if at all possible. Equipment removed from the site to the regional (district) office for repair or calibration should be returned to the site in less than ten working days. All corrective actions are to be recorded in the affected instrument's log book.

4.1.3 Sample Collection and Handling

Sample collection and handling refers to all procedures carried out by regional personnel from the time the cleaned, prepared cartridges are received from the laboratory to the time the collected sample is packaged and shipped back to the laboratory for analysis. The objectives of these procedures are two-fold: i) to collect, handle, and transport the samples with no effects to their physical or

chemical integrity, and ii) to obtain all necessary information related to the collection of the samples. A number of QA requirements and activities have been incorporated into the various stages of collection and handling to meet these objectives.

i) When installing or removing sampling cartridges from the manifold, cotton gloves are to be worn to minimize chances of contamination.

ii) Cleaned cartridges received from the lab are to be stored in a dedicated refrigerator until one hour prior to installation for sampling.

iii) Sampled cartridges are to be stored in a dedicated refrigerator until such time as the paperwork is completed and the sample is shipped to the lab for analysis.

iv) Coolers fitted with freeze-packs are to be used for shipping cartridges between the laboratory and the regional sites.

v) A sample submission/history form is to be completed for each submission; the original is to go to the lab with the sample, a copy is to be sent to the network coordinator, and the region is to keep a copy.

vi) Sample contamination is to be minimized through periodic cleaning of the glass sampling inlet and the sampling manifold as required.

The previously outlined QA activities are by no means all-inclusive. The Field Operations component is closely linked to other QA activities such as routine special studies and non-routine special studies and performance and systems audits; all of which are to be used to evaluate the network performance and to ensure that procedures are well conceived and scientifically sound. The following discussion describes only routine special studies. Non-routine special studies and systems and performance audits, which are sporadic in nature, will be described in an upcoming section entitled "Other Network QA Considerations".

Routine special studies to assess precision and accuracy in the Field Operations are to include:

i) Collocating air samplers for a fixed period of time on a rotating basis at least one sampling site per region.

ii) A handling blank is to be submitted quarterly from each site.

iii) A travelling blank is to be submitted with each set of samples.

4.2 Laboratory Operations

The co-ordination of the analysis of volatile organic compound samples is the responsibility of the Air Unit of the Trace Organics Section of the Laboratory Services Branch, located in Rexdale. The three major functions of the laboratory operations are i) to provide clean cartridges on a routine basis; ii) to perform the analysis of and report the results from the VOC samples generated by monitoring activities, and iii) to support the field operations through routine quality control procedures and special studies activities.

To meet these objectives, the laboratory must follow stringent quality assurance and internal quality control[1] procedures (which are to be documented in the QA Manual) to produce data of known precision, accuracy, and comparability. A number of QA considerations are implied:

i) Specific quality control and quality assurance procedures are to be designed, documented, and carried out to determine the precision, accuracy, and comparability of the laboratory results. Acceptable limits of precision, accuracy, and comparability are to be defined and

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[1]'Internal quality control' is defined as "The routine activities and checks, such as periodic calibrations, duplicate analyses, use of spiked samples, included in normal internal procedures to control the accuracy and precision of a measurement process." (U.S. EPA, 1976)

documented.

ii) 'Function' and 'control' checks (defined below) are to be systematically performed on both a real-time and long term basis to ensure that acceptable operating conditions are maintained and to identify situations which do not meet QA objectives. Corrective action procedures are to be followed to rectify 'out-of-control' situations.

iii) The laboratory must produce data within a reasonable time frame after receipt of the samples and in a format suitable for subsequent data manipulation, screening and analysis.

The elements of laboratory quality assurance which can directly affect the precision, accuracy, and comparability of the volatile organic compound data are;

i) Sample analysis

ii) Laboratory sample handling

iii) Support of field measurements

4.2.1 Sample Analysis

The determination of volatile organic compounds is accomplished by thermal

desorption, cryogenic pre-concentration, and subsequent analysis by dual capillary column gas chromatography. The desorber/pre-concentrator is a 10 port unit custom-built by Metrex; the gas chromatograph is a Hewlett Packard 5880 with a DB-1 (non-polar) column and a DB-5 (slightly polar) column.

These instruments are to be operated according to their design specifications outlined in their instrument manuals (which are to be kept on file with the QA Plan and QA Manual). The Metrex desorber presents a problem as it is a custom-built machine that has undergone extensive modifications to get it to function properly. As problems are encountered and corrected they should be fully documented so that a record of the instrument is compiled for future users.

The systematic application of quality control procedures on both a real-time and long-term basis is required to evaluate data quality. These procedures consist of i) function checks which verify the sample validity at the time of analysis and confirm that the instrument or analytical procedure was operated correctly and under proper conditions; and ii) control checks which provide a measure of the variability of the results obtained in terms of precision and accuracy and identify deteriorating or out-of-control conditions as they develop to ensure that they are rectified immediately.

Function checks are to be performed at the analyst level and are to include:

i) Correct storage of the sample prior to analysis. This entails storing samples in a refrigerator until 1 hour before they are to be analyzed.

ii) Verification of the samples' integrity. The transport end caps must be securely attached; the cartridge must be free from cracks and broken ends; and there should be no information stickers of any sort attached to the cartridge itself.

iii) Ensuring that the analytical equipment is working prior to operation. All instrumentation is to be properly warmed up and instrument drift and noise are to be checked and documented.

iv) Adjustment for zero, span, and operating ranges if necessary.

It is important that the system be reliably maintained through proper service and maintenance procedures to minimize and quantify the variability observed between runs.

Control checks are to be performed by the analyst as part of the routine analysis procedures and by laboratory QA personnel without the knowledge of the analyst. The selection of the type of control checks to be used and their frequency of use is to be determined by the Laboratory QA Manager for each analytical

procedure. Control checks are to consist of:

i) Instrument calibrations, which are performed to identify the instrumental response to known concentrations of organic compounds in order to relate instrument response to the corresponding concentration of unknown samples. If a calibration can not be confirmed, corrective action must be undertaken (i.e. re-calibration) to bring the calibration back into control before the analytical run commences.

ii) Submission of QC samples ('blind' samples, laboratory blanks, or real VOC samples) at various stages of the analytical stream is required to assess the precision and accuracy of the system as well as the detection of analytical bias or introduction of contamination. Once again, the selection of the type of control samples to be used and the frequency with which they should be analyzed is to be determined by the Laboratory QA Manager.

iii) The results of the analysis of QC samples are to be plotted on a real-time basis against historically determined statistical limits for "warning" and "out-of-control" situations. This is done by calculating the mean (x) and standard deviation (σ) of a number of repeated

measurements. "Warning limits" are to be set at $x \pm 2\sigma$ and the "out-of-control" limits at $x \pm 3\sigma$. Warning and out-of-control limits are to be periodically recalculated to include recent measurements in the statistical calculations. Results that exceed the control limit values are to be appended with an appropriate lab remark code.

iv) The Air Unit of the Trace Organics Section is to participate in VOC interlaboratory round-robins to assess accuracy and comparability.

v) The validity of the results produced by the Air Unit of the Trace Organics Section is to be assured. This requires checking samples to see if they are identified and labelled as described of the Sample Submission/History Form. This check is to be done before samples are entered into the analysis queue. The laboratory supervisor must screen all data sets before releasing the data to the Sample Information System (SIS) to ensure that all requested analyses were performed, that data transcription errors or omissions had not occurred, and that unreasonable or obvious errors were not recorded in the data sets.

4.2.2 Laboratory Sample Handling

The potential for sample contamination, mishandling, or degradation within the laboratory is to be kept to a minimum by following established sample custody and sample storage procedures. Sample custody procedures are needed to ensure the progression of all samples through the laboratory analysis stream from sample reception, storage of the sample prior to analysis, and the analysis itself. A system for monitoring and recording the sample custody stages within the laboratory is to be implemented and is to involve the use of a sample custody form (see QA Manual). This form is to include the following information: sample identification; receiver's signature and comments; date entered into the laboratory system; analyst identification; and the date that all analyses were completed.

Specific activities for sample custody and sample storage are listed below:

i) Upon receipt at the laboratory, all cartridges are to be visually inspected for cracks and breaks and to ensure that they are properly labelled and arrive with supporting documentation.

ii) Laboratory sample custody procedures including logging the samples into the laboratory information system and the issuing of a sample custody form are to be initiated.

iii) Supporting documentation (Sample Submission/History Form) is to be filed and kept on hand for easy reference.

iv) Samples are to be stored at 5°C in a refrigerator dedicated to VOC samples.

v) Corrective action procedures required as a result of problems observed at the sample reception stage are to be done.

vi) A reasonable time limit is to be established for the analysis stage to ensure that sample analyses are completed before significant degradation of the sample occurs.

4.3 Data Management Operations

The function of data management operations is to compile, screen and validate, transfer, report, and store, correctly and efficiently, data generated by the Volatile Organic Compound Sampling Network. The data management system is broken down into the following modules:

i) Data collection and entry

ii) Data management

iii) Data screening and validation

iv) Database structure

v) Data reporting

vi) Standard data analyses

vii) Provision of data to external users

The data management system and its associated QA activities are shown in Figure 4.1.

4.3.1 Data Collection and Entry

The data collection and entry system provides the means for transferring the sampling and analytical information from the field and laboratory into the preliminary (working) database as quickly and efficiently as possible. All relevant information is to be reported at each stage of the process and errors in data recording and transfer are to be minimized through the use of standardized data coding forms, transmittal procedures, and data entry procedures.

Data collected at the field operations level are to be documented on the Sample Submission/History Form (see S.O.P. and Technical Manual). This form combines the LIS Submission Form with the Request for Analysis Form and also provides space for recording field sampling information. The following sampling

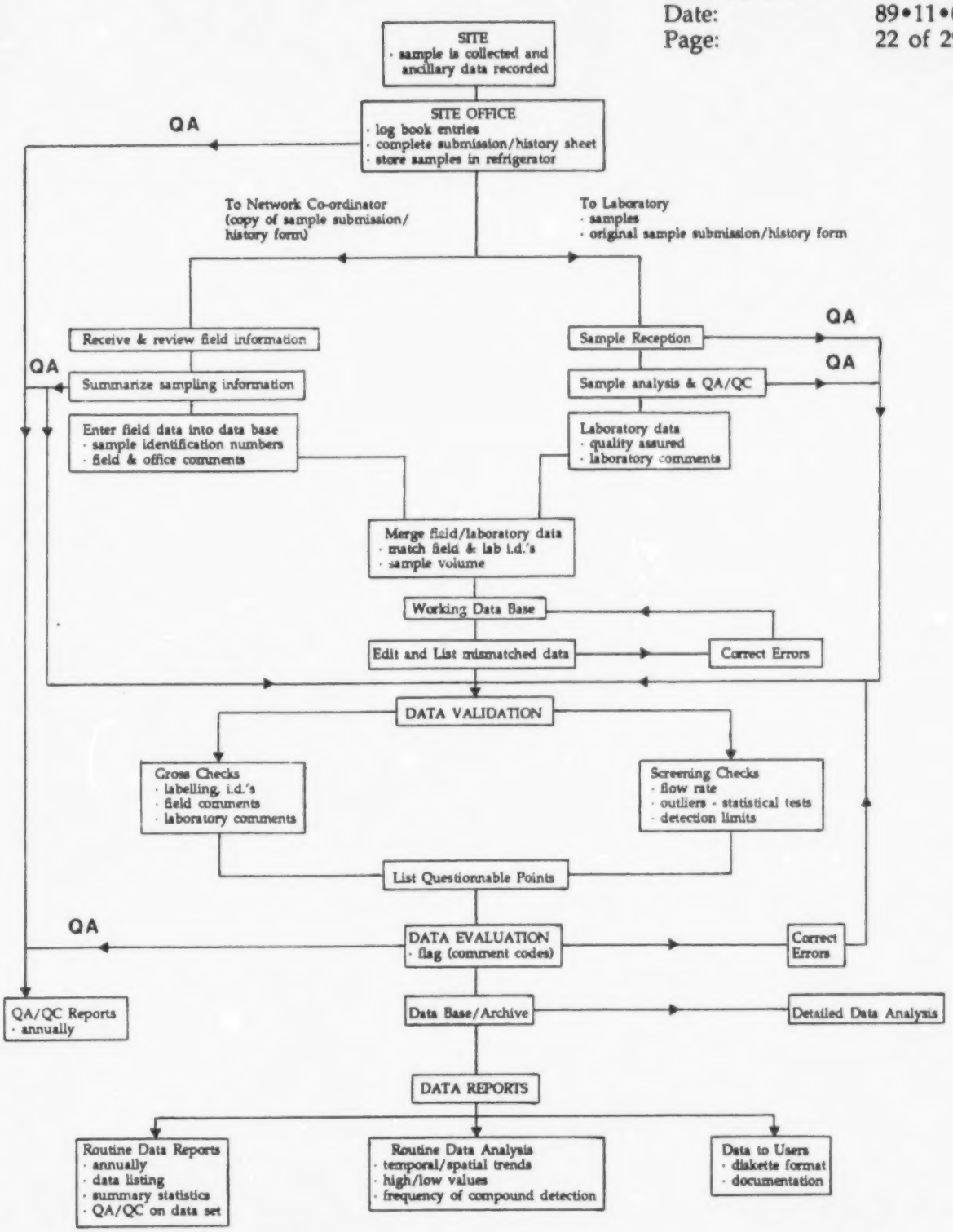

Figure 4.1. Network Data Handling Structure.

information must be recorded: submission number; date submitted; region in which the sample was taken; telephone number of the primary contact in that region; client codes of people to whom copies of the LIS report should go; the name of the field operator; and the name of the person submitting the sample. In addition, the following sample history information must be provided: sample number; date the sample was taken; station identification number; the start and end readings from the elapsed time counter; total minutes sampled; flow rate; volume sampled; field comments, i.e. any factors which may have influenced the integrity of the sample such as instrument problems, cartridge breakage, non-standard sampling period or time etc.; and special remarks, a section used to elaborate on field comments. The original copy (in green ink) of the completed form is to accompany the samples to the laboratory. A copy is to be retained by the regional technician for easy reference and a second copy is to be sent to the Network Coordinator, Air Resources Branch.

The quality assurance procedures associated with the collection of field data consist of ensuring that i) the regional technicians are properly trained in the data recording procedures; ii) they record all pertinent information; and iii) periodic evaluations of the system used to record and transmit the information are carried out.

The objective of the laboratory data management system is to provide a

quality controlled set of analytical results that can be merged with the field sampling information for subsequent data screening and validation. A number of preliminary QA checks are to be done before the analytical data are released from the Laboratory Information System (LIS) into the Sample Information System (SIS). These checks include i) verifying sample labelling and matching field sheets with samples; ii) ensuring that an analytical record exists for each sample described on the field sheets; iii) checking for obvious transcription errors; iv) checking the laboratory data printout against original field sheet information; and v) ensuring that any missing or coded results are explained.

4.3.2 Data Management

The management of the volatile organic compound database is the responsibility of the Network Coordinator, Air Resources Branch. Management of the database includes:

i) creating a working database;

ii) entering field data into the database;

iii) merging and editing field and laboratory data;

iv) data screening and validation;

v) final data archiving;

vi) data reporting.

This segment will discuss data management up to merging and editing field and laboratory data. The other aspects of data management are discussed in detail in sections 4.3.3 to 4.3.7.

The working database is to be developed and maintained at Air Resources Branch in one of the major database languages available such as dBASE IV or PCFOCUS. Analytical results from the SIS are to be down-loaded to the working database where all data management operations are to be carried out. When the data is in final form it will then be transferred to the Air Quality Information System (AQUIS) which is to be the final data repository.

The field information sheet forwarded to the Network Coordinator is to be scrutinized for incomplete data entries and labelling errors and legibility. This preliminary review at the data reception stage allows for the correction of omissions and errors in the Sample Submission/History Form while the regional technician is still familiar with the submission.

The working database at ARB is to be used to merge field and laboratory data. A program is to be developed to match the field and laboratory data for all samples. The program is to list samples which do not have matching field and laboratory data and must have the capability of correcting and editing the working database. After all data have been matched and all sample identification errors and

omissions have been corrected the working database can be subjected to data screening and validation programs.

4.3.3 Data Screening and Validation

Data screening and validation techniques are to include automatic (computerized) procedures designed to identify and evaluate questionable data (these data screening and validation techniques are documented in the QA Manual). The computer data screening and validation procedures are to be used to compare all data to a set of predetermined standards (e.g. standard sampling period or sampling time, volume checks, gross limit checks, etc.) and to list any data which do not conform to the prescribed screening and validation criteria. Based upon these results the data will either be objectively accepted as stated or accepted with qualification. If the latter is the case, the affected record will be appended with an appropriate comment flag. At this point in the data management operations no data will be rejected (invalidated) from the database.

4.3.4 Database Structure

For quality assurance purposes, the data archiving system must provide means of ensuring that a complete, correct data set is maintained, that it is secure

(i.e., restricted access for editing) and that it has the capability of retrieving QA/QC information for all data collected. The database must be in a standardized format such that data access and manipulation routines may be developed to extract and sort subsets of data for data analysis purposes as well as for generating data tapes or diskettes for authorized users. Inherent with the archiving system is the need to restrict access to editing and deletion routines to authorized personnel only.

4.3.5 Data Reporting

Data reports are to consist of volatile organic compound concentrations, annual data summary statistics, and QA/QC data summaries. Data is to be released within the Ministry in report form only after its quality has been assessed and documented. The data listing and annual summary statistics reports are to be issued yearly, within nine months of data collection. QA/QC data summaries are to be released bi-annually.

All reports are to be in a format that is easily readable. For example, the field information and data for each parameter for a particular site are to be condensed onto as few pages as possible; column titles are to indicate the parameter being reported and the units of measurement; and all remark codes are to be clearly explained. All reports are to include site descriptions and descriptions

of analytical limits of quantification.

4.3.6 Standard Data Analyses

Standard data analyses are the responsibility of the Network Coordinator of the Air Resources Branch and are to include calculations of daily, seasonal, and annual concentrations and data distributions for all species and on an individual site, region, and network basis. Collaboration between ARB and the monitoring regions will determine which data are used in the data analyses. The exclusion of unreliable data must be based on objective rejection criteria (e.g. severity of field comments).

More detailed data analyses are to be done by both the Air Resources Branch and the monitoring regions after the data verification process has been completed.

4.3.7 Provision of Data to External Users

Screened and validated data are to be made available to external users (i.e., non-Ministry agencies) upon demand, either in printed report form, on magnetic tape, or on diskette. The external users are to receive a well-documented description of the data file format and contents (parameter format, units of each

parameter and description of remark codes) to facilitate easy extraction. The documentation is also to include a synopsis of the network operating procedures and QA/QC procedures, site descriptions, data screening and validation techniques, a description of the analytical limits of quantification, a summary of the network's annual data summary statistics (e.g., range of values, seasonal averages for each parameter, etc.) and a disclaimer stating that it is up to the discretion of the user to include or delete questionable data in their working database (since the data provided to external users is only qualified, not invalidated).

5.0 Other Network Quality Assurance Considerations

The QA considerations to be presented in this section fall outside the considerations of previous sections or extend beyond one section into several. All have been peripherally mentioned yet warrant more detail. Other network QA considerations are:

i) Training and upgrading;

ii) Corrective action;

iii) Network documentation and document control;

iv) Non-routine special studies;

v) Systems and performance audits;

vi) Quality assurance reporting.

5.1 Training and Upgrading

It is of the utmost importance for network personnel to be updated and upgraded in their training to account for changes and/or additions in their QA responsibilities. This is accomplished in the following manner:

i) All network personnel are to have a complete set of the Standard Operating Procedures and Technical Manual on hand for quick, easy

reference. If a new procedure is to be implemented in the network, it is the responsibility of the Network Coordinator to update and distribute copies of this procedure for inclusion in the manual, as described in Section 5.3 (Network Documentation and Document Control).

ii) The regional technician is to become familiar with each new procedure and must be capable of performing it. If necessary, the Network Coordinator is to observe the regional technician perform this procedure to ensure it is done correctly.

iii) Unless required as an emergency measure, new operational procedures are to be implemented only at specified times throughout the year, usually after "technician" meetings where the new procedures have been explained. Such consolidation of changes helps to prevent haphazard implementation and ensures that all network personnel are receiving consistent training and upgrading.

iv) Regular communication is to occur between the Network Coordinator and the Air Unit Trace Organics Section Supervisor and the senior regional technicians to ensure that the operating system is progressing smoothly and that notification of any problems is promptly served

and acted upon.

In addition to providing training procedures, "technician" meetings allow for communication and feedback to take place. These meetings are to be held annually at the Air Resources Branch and are to be attended by the Network Coordinator, senior regional technicians, and the regional technicians responsible for the operation of sampling sites.

Training inadequacies or unsuitable operating procedures are sometimes detected during routine supervisory checks or during external QA audits. It is the responsibility of the Network Coordinator to bring these inadequacies or procedural problems to the attention of the regional Air Quality Assessment Chief who is to act upon the problem. If necessary, the Network Coordinator is to offer any technical assistance needed to correct problems identified.

The discussion above is relevant to personnel already in the network. When new personnel enter the network, initial training and follow-up training is to be done. New technicians are to become familiar with all network documentation and are to receive thorough training under the supervision of the senior regional technician.

5.2 Corrective Action

Inherent with a monitoring program are sampling problems which may result in lost, inaccurate, or imprecise data. Most problems, such as instrumentation failure, can be dealt with using an immediate corrective action scheme. Systematic problems, however, are often detected only after non-routine special studies or system and performance audits have been conducted. In either situation, to ensure that problems of non-conformity within all network components are detected, eliminated, and reported on in a timely manner, a "closed-loop" corrective action scheme for both short- and long-term problems should be followed. This entails:

i) define the problem;

ii) assign responsibility for investigating the problem;

iii) investigate and determine the cause of the problem;

iv) determine the corrective action to eliminate the problem;

v) assign and accept responsibility for implementing corrective action;

vi) establish the effectiveness of the corrective action and implement the correction;

vii) verify that the corrective action has eliminated the problem.

All corrective action plans are to be documented in the QA Manual. For minor problems, it should only be necessary to consult checklists, control charts,

trouble-shooting guides, and instrument manuals for the corrective action to be performed on-the-spot. If the problem is serious enough to affect data completeness or data accuracy (i.e., it can not be fixed in the field), the problem should be corrected at the regional (district) office. No more than 3 sampling days should pass before the problem is rectified and sampling resumes. The success of the corrective action scheme hinges on the early detection of the problem and the response time needed to correct the problem. Long-term corrective action requires that the sequential, methodical approach be applied to systematic problems because they can have a substantial impact on the network's data quality as a whole. A Corrective Action Log Form (see QA Manual) should be completed by the Network Coordinator for each non-conforming situation as it is identified.

5.3 Network Documentation and Document Control

Documentation necessary for the proper operation of the network is essential and must be properly produced, updated, and distributed on a regular basis. A document cataloguing system, which lists document type, document identification number, title, author(s), date of publication, date(s) of revision(s), and a user distribution list is to be maintained by the Network Coordinator. This listing is to be incorporated into the QA Manual.

Any changes in operating procedures are to be immediately updated and

distributed to all personnel. The page indexing format (see, for example, the top right corner of this page) is to be used for easy insertion of revised, updated, or additional pages into existing documents. Each major section should begin on a new page so that the revision of single sections can be done without affecting other sections. When new pages are revised, they are to be substituted for the old and a notation is to be made in the Table of Contents.

5.4 Non-Routine Special Studies

Additional or modified operating procedures are to be initiated on a non-routine basis to investigate and quantitatively assess site-specific, operation-specific, or network-wide phenomena or peculiar data trends as they affect precision, accuracy, completeness, representativeness and comparability. These "special studies" may also be considered a part of long-term corrective action or as a follow-up to systems and performance audit recommendations.

Special studies are to be initiated as soon as possible after the need has been identified, but only after the Organics Monitoring Sub-Committee has given approval. Special studies must be carried out under the strictest quality control and quality assurance procedures. As such, each special study must be documented in terms of its quality assurance activities and structure. In essence, each special study is to have a *condensed* QA Plan composed of:

i) a description of the study and its objectives;

ii) a project schedule;

iii) an outline of responsibilities;

iv) a description of the sample collection, handling, and analysis procedures, data screening and validation procedures, and QA/QC procedures specific to the special study;

v) a data reporting scheme.

5.5 Systems and Performance Audits

Audits to assess the quality of the data and the collection and measurement processes are to be carried out on a routine basis by auditors independent of the monitoring network. These audits are to consist of systems and performance audits to determine qualitatively and quantitatively i) whether the quality of the network's operations comply with desired standards of operation, accuracy, precision, representativeness, completeness, and comparability; ii) whether the existing operations are sufficient to maintain those standards; and iii) whether the Volatile Organic Compound Monitoring Network's QA program is satisfactory for assuring the standards. To meet these objectives, an external auditor is to evaluate the representativeness of the sites in the network (50% of the sites per audit), assess the collection and measurement systems of the network and assess the data management system. External audits are to be done bi-annually if they are deemed

warranted by the Organics Monitoring Sub-Committee. Annual internal performance audits of the Field Operations component are to be done by the Instrumentation Unit of the Air Resources Branch and the performance of the Laboratory Operations component is to be assessed by the Quality Assurance Office of the Laboratory Services Branch.

The external auditor chosen to carry out the audits should be experienced and knowledgable in the fields of quality assurance and volatile organic compound monitoring. The design of the audit depends upon the guidelines given in the QA Plan. A specific plan for the audit is to be produced by the auditors and is to be evaluated by selected members of the Organics Monitoring Sub-Committee before it is carried out to ensure that the plan meets the needs of the network. A final report is to be prepared by the auditors and submitted to the Organics Monitoring Sub-Committee upon completion of the audit. The report is to contain the results of the systems and performance audits including details of the site evaluations, technician assessments, and the results of the evaluation of the data management system. Weaknesses, strengths, and recommendations generated by these evaluations are to be reported in detail. Any previous audit recommendations are to be followed up by the auditor to determine if these recommendations were implemented, and if not, why they were not implemented. A synopsis of audit results is to be incorporated into the annual QA reports prepared by the Network Coordinator.

Since external audits are essential to the success of the QA Program and the Volatile Organic Compound Monitoring Network in general, they warrant further elaboration. The requirements to verify, examine, and evaluate that site representativeness and the collection, measurement, and data management components have been properly documented and effectively implemented in accordance with specific objectives are given below.

To assess site representativeness, the accuracy and completeness of current site documentation must be evaluated. The auditor's check for accuracy and completeness of site documentation must be carried out in conjunction with site inspections. Inspection of the site in relation to its description will determine if the documentation is inaccurate or incomplete and will serve as a check on the regional technicians to ensure they know the procedure for reporting site changes. Site inspections also evaluate the site in relation to the network's siting criteria. This evaluation must be a value judgement by the auditor, either in the form of a relative grading (e.g., excellent, good, marginal, unacceptable) or a stated opinion on the adequacy of the site for measuring VOCs representative of the surrounding environs. Site evaluations are to be documented in the auditor's final report to the Organics Monitoring Sub-Committee. If sites are rated marginally acceptable or unacceptable by the auditor, the Organics Monitoring Sub-Committee must consider corrective action, such as, i) leaving the site as is but documenting the reasons for its acceptance; ii) upgrading the site by making physical changes to it;

iii) initiating special studies at or near the site to assess quantitatively the magnitude of the problem; and iv) failing all else, relocate the site. It is the responsibility of the Network Coordinator to act upon measures recommended in the auditor's report as well as updating site documentation, assessing the effects of the offending site on data representativeness and to determine why the differences were not detected as part of routine network operations.

The Field Operations component must also have its instrumentation and sample collection and handling procedures audited. The systems audit on this portion of the network operation is to consist of the following:

i) assessment of the completeness and accuracy of the Standard Operating Procedures and Technical Manual in relation to field instrumentation requirements and procedures and sample collection and handling procedures;

ii) assessment of the design, adequacy, and effectiveness of the procurement and pre-testing, inventory control, calibration, preventive maintenance, and routine checking and corrective action procedure;

iii) assessment of the adequacy of the network's instrumentation for meeting the network's overall objective and the network's quality

assurance objectives with respect to accuracy, precision, completeness, representativeness, and comparability.

The performance audit would consist of a quantitative assessment of the field instrumentation, namely:

i) on-site calibrations of all instruments;

ii) measurement of on-site operating conditions.

The major emphasis of external laboratory audits is placed on evaluating the laboratory's QA/QC procedures and their effectiveness in terms of meeting the network's requirements for quality assurance rather than a detailed investigation of each of the specific components within the laboratory system. As such, the audit will concentrate on evaluating the documentation of current operating procedures, QA/QC procedures and the QA responsibilities of the laboratory personnel involved.

Auditing of the data management component is limited in scope. The systems audit portion will concern itself with an assessment of the design, adequacy, and effectiveness of the data screening and validation techniques used to flag data, if required. The effectiveness of the data screening and validation

techniques is to be evaluated in a performance audit by, i) performing spot checks on calculations used in the screening and validating process; and ii) inserting "dummy" data (unknown to the Network Coordinator) into the database, performing the screening and validation checks on them, and determining if the appropriate qualifying remark codes are appended to these records.

All findings and recommendations of the external auditor's systems and performance audit are to appear in a final auditor's report to the Organics Monitoring Sub-Committee, which is to evaluate and act upon the report's recommendations, where appropriate and feasible.

5.6 Quality Assurance Reporting

Annually, a QA Report is to be prepared by the Network Coordinator summarizing the results of the QA Program over the preceding twelve-month period. This document is to report any information which will help determine the confidence that users may place in the data, the level of QA applied to the network, its effectiveness, and recommendations on QA-related problems. This report is an effective means of evaluating the costs of network quality assurance in terms of the increased data quality. The annual QA Report is to be presented to the Organics Monitoring Sub-Committee for review and corrective action.

QA reports are to include the following specific information:

i) changes made to the QA Plan, QA Manual, or the Standard Operating Procedures and Technical Manual;

ii) measures of data quality, namely:

a) statistics of sample and data capture (completeness) and reasons for lost data;

b) results of blank, replicate, blind, and collocated sample analyses (precision and accuracy);

c) laboratory QC data (precision, accuracy, comparability);

d) results of special studies (precision, accuracy, comparability);

iii) significant QA problems, accomplishments, recommendations, and corrective action to be taken;

iv) a synopsis of internal and external systems and performance audits;

v) status of data, including projected schedules for data reports;

vi) summary of QA training.

Upon review, QA reports are to be distributed to all network personnel and to all data users on the documentation distribution list.

6.0 References

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